

Direct Separation of Short Range Order of Intermixed Nanocrystalline and Amorphous Phases

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Beamline: X16C

Introduction: Diffraction Anomalous Fine Structure (DAFS), EXAFS and XRD measurements have been combined to separately determine short-range order (SRO) about a single atomic type in each phase in a sample of mixed amorphous (a) and nanocrystalline (nc) phases. We applied this method to study the local structure of mixed phase samples containing a- and nc- Ge supported by an SiO₂ matrix. EXAFS yields information about the SRO of all Ge atoms in the sample (a-Ge and germanium oxide), while DAFS determines the SRO of only the ordered (nc-Ge) fraction. Our approach can be applied to many systems of mixed amorphous and nanocrystalline phases.

Methods and Materials: The sample was prepared by co-deposition of Ge- and Si- oxides onto quartz substrates by radio-frequency magnetron sputtering and by a subsequent annealing at 800 °C for 1h. All three experiments, XRD (Fig.1), DAFS (Fig. 2) and EXAFS (Fig. 3) were performed at room temperature using a custom-designed 4-circle Kappa diffractometer.

Results: By comparing the DAFS results obtained for the nc-Ge phase only with EXAFS results, obtained for the mixture of a-Ge and nc-Ge (and Ge oxide), we determined that the first-shell distance distribution is bimodal: the nanocrystalline distance is the same as the bulk crystal, to within 0.02 Å, but the mean amorphous Ge-Ge bond length is expanded by 0.08(2) Å. We interpret this elongation as caused by the interfacial disorder at the SiO₂ boundaries.

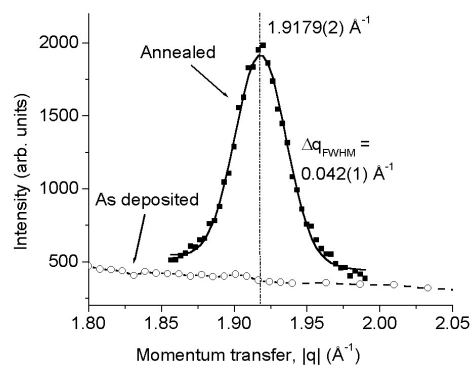


Figure 1: XRD measurement of Ge (111) peak.

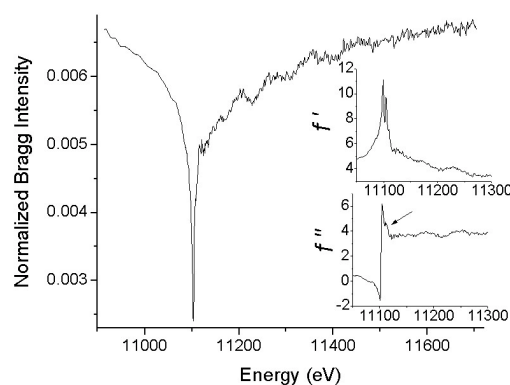
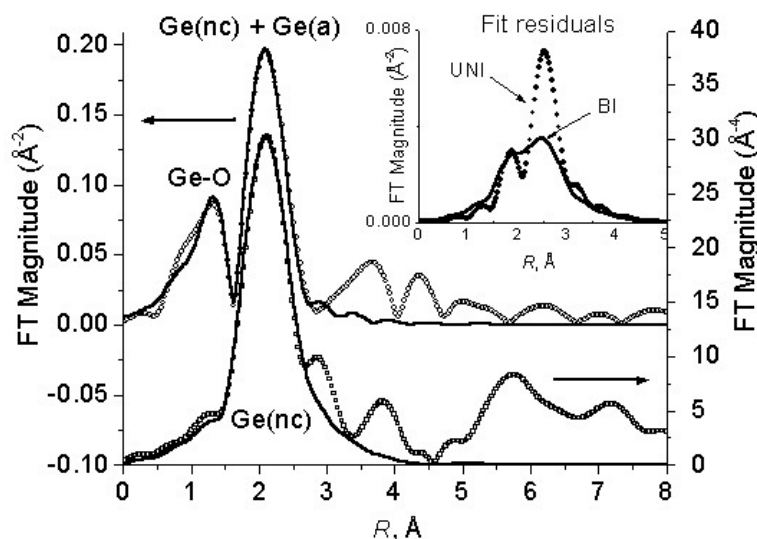
Figure 2: Raw DAFS and separated f' and f'' data.

Figure 3: Fourier transform magnitudes of FEFF7 theory (solid) and the data (symbols). Top left: Mixture of Ge oxide and (a+nc) Ge as obtained from EXAFS. Bottom left: fit to the data extracted from DAFS (nc Ge contributions only). The inset shows fit residuals after fitting the data with the unimodal (UNI) and bimodal (BI) models of Ge-Ge distributions.